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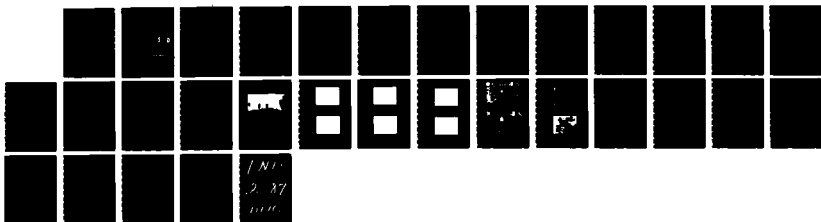
RECLASSIFICATION AND GREASE COMPATIBILITY STUDIES FOR
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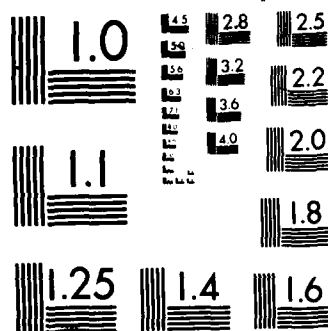
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**RECLASSIFICATION AND GREASE COMPATIBILITY STUDIES
FOR LIQUID PROPELLANTS**

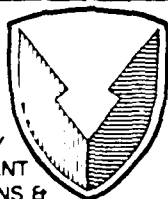
WILLIAM J. CRUCE

**HAZARDS RESEARCH CORPORATION
200 E. MAIN STREET
ROCKAWAY, NJ 07866**

**WILLIAM O. SEALS
PROJECT ENGINEER
ARDEC**

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20. ABSTRACT (cont)

change occurred in the shock sensitivities when compared to the results of the neat liquid propellant formulation. Thermal stability test results showed NOS-365 was unaffected when mixed with Molykote 33, but reacted at significantly lower temperatures with Fel-Pro C100, Molykote M-77 paste, and Molykote FS 3451. A mixture of Fel-Pro C100 grease with LP 1845 was significantly less thermally stable than neat LP 1845; while Molykote 33, Molykote M-77, and Molykote FS 3451 had little effect upon the thermal stability of the liquid propellants. *See table 2-10*

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INTRODUCTION

Liquid propellant formulations must be shipped as "Class B" explosives.¹ When this classification was established, the packaging configuration or quantity levels of material were not taken into consideration. In 1979, Hazards Research Corporation performed experimental tests² on NOS-365 and LP 1845 in accordance with the tests specified in the Department of Army Bulletin Cl, TB-700-2, Chapter 3. In this earlier work, the results demonstrated that the liquid propellant formulations were considered Class 7 (explosive) when under heavy confinement. When these same materials were placed in a glass or plastic container designed to vent product gases at low pressures, they were considered Unrestricted (not explosive). On the basis of the detonation and card gap tests, a Class 2 was recommended for these materials. However, the ability of a sample to propagate or detonate is influenced by the degree of confinement and critical mass. Classification of small lot sizes of liquid propellant in low confinement packaging as a Class 2 material cannot be based upon experiments performed in a steel pipe under high confinement. The work effort of this program was to determine if small sample sizes of liquid propellant packaged in low confinement containers might be more properly classified at a lower level.

In addition, Hazards Research Corporation has information to indicate that NO_2 and N_2O_4 plus silicone grease mixtures can sustain a detonation. Since various silicone greases come in contact with the liquid propellants in the gun test fixture, a potential source of ignition could develop from a decomposition reaction. Accordingly, this study was designed to provide basic information on the sensitivity of mixtures of silicone greases with these liquid propellant formulations.

RECLASSIFICATION OF LIQUID PROPELLANTS

To establish the sensitivity of NOS-365 and LP 1845, detonation tests were conducted that used the constant current resistance technique. A 500 ml polyethylene container, designed to vent product gases, was initially filled with water. Through the top of the filled container was placed a detonation velocity probe. A 160 gram RDX (cyclotrimethylene trinitroamine) booster charge positioned at the bottom of the container was ignited. To produce the effects created by the liquid propellants, the test was repeated with a simulant water/salt mixture having a density of 1.34 g/cm^3 . The distance-time trace for the water/salt mixture would serve as the standard for determining whether a detonation occurred. A comparison was then made

¹Letter, Subject, "Approved Hazard Classification of NOS-365 Monopropellants; notice of," from J.J. Duffy, Jr., Commander, Naval Ordnance Systems Command, to Hdq. Military Traffic Management and Terminal Services, 24 December 1973.

²Cruice, W., "Classification of Liquid Gun Propellants and Raw Materials For Transportation and Storage", Hazard Research Corporation, Contractor Report ARBRL-CR-00454, May 1981.

of the distance-time trace for water/salt mixture and each of the propellants tested.

A test with an NOS-365 filled container was conducted. If no detonation occurs, then an NOS-365 filled 500 ml container is placed on a 160 gram RDX booster in the bottom of a standard shipping carton (DOT-12B, No. 275 double-wall fiber carton). This shipping carton complies with DOT regulations for the shipment of oxidizing or corrosive materials. To absorb spilled liquid propellant and lend support to the shipping carton, vermiculite³ was added to the void space between the carton wall and the container. A 4"x4"x0.375" cold-roller steel witness plate is placed on the top of the NOS-365 filled container and surrounding vermiculite. The witness plate is immediately beneath the top of the carton when it is closed. After the entire package is assembled, a detonation-velocity probe is passed through the witness plate as shown in Figure 1.

The assembled package (donor) was placed in the center of four similar packaged NOS-365 cartons (acceptors) spaced at right angles of 12, 18, 24, and 36 inches (illustrated in Figures 2 and 2a). These similarly packaged cartons of liquid propellant were without donor charges, detonation velocity probes, or witness plates. The nearest acceptor unit was equipped with a section (96 inches) of Detacord⁴ that extended from the plastic 500-cm³ container to a section of lead sheet that was wrapped around the detacord. This would act as an independent witness to any propagating detonation in the acceptor. Any detonation occurring in the acceptor would produce a detonation of the PETN-filled Detacord. Damage to the lead sheet would then be physical evidence that a detonation had occurred.

RESULTS OF THE EXPERIMENT

The results of the detonation tests are summarized in Table 1. As shown here, the detonation-velocity probe indicated a decaying reaction took place in each of the test. This is confirmed from the distance-time traces (Figures 3 thru 8) for each of the tests. In the packaged array configuration test for NOS-365 and LP 1845, shown in Figures 7 and 8, notice that only a slight variation occurred in the decay rates between each of these materials. When compared to the simulant salt/water mixture decay rate, the NOS-365 and LP 1845 decay rates were faster.

The reaction in the packaged configuration yielded enough energy to fracture the witness plate, but did not produce a clean hole indicative of a detonation. These results are shown in Figures 9 and 10. In both array configurations of NOS-365 and LP 1845, there was no indication of an explosive reaction in any of the four acceptors. As seen in Figures 9 and 10, all the plastic acceptor containers were recovered from the NOS-365 and LP 1845 tests. The acceptor container positioned nearest the donor in both

³Vermiculite is the recommended filler material but contains 5% iron oxide which reacts with the liquid propellants.

⁴ Ensign-Bickford "E" cord, 25 grains PETN/ft. O.D. 0.162 in.

tests, was more charred than the rest, especially for the LP 1845 test. The Detacord and lead sheet elements were recovered without damage. All the donor containers were destroyed by the energy released from the donor charge and decomposition of the primary sample. The results of the witness plate damage is found in Figures 11 and 12. In all the witness plates examined, there was no evidence of an explosion.

SENSITIVITY OF LIQUID PROPELLANTS WITH SILICONE GREASES

Description of Experiments

Task 1: Trauzl Block Test

The following four silicone greases were selected for evaluation:

1. Molykote 33 (medium consistency silicone grease) Dow Corning, Midland, Michigan 48640.
2. Molykote FS 3451, Fluoro-Silicone, Dow Corning, Midland, Michigan 48640.
3. Molykote M-77 paste Dow Corning Cat. No. 85450-41, Dow Corning, Midland, Michigan 48640.
4. Fel-Pro C-100 Hitemp Antigall Part No. 51017 Fel-Pro Inc., Skokie, Illinois 60076.

Sample mixtures were made of each grease with NOS-365 and LP 1845 (total of 8 sample mixtures). Each sample mixture consisted of 2 grams of liquid propellant with 2 grams of one of the four greases. These mixtures were hand blended and stored for 24 hours at ambient temperatures prior to use.

The Trauzl Block test was used to measure the explosive power of a sample material. In this test, a glass vial containing a weighed quantity of the sample was placed in a lead cylinder (1/2 inch thick wall). A No. 8 blasting cap was electrically activated and a volume increase of the lead cylinder caused by any propellant reaction was measured. The specific expansion in cm^3/g is calculated from the volume increase from the test sample less that obtained from a blank run divided by the mass of the sample. Water was used in the blank test run.

Task 2: JANAF Thermal Stability Test

The JANAF thermal stability test is the standard test designed by the Interagency Chemical Rocket Propulsion Group for testing the thermal sensitivity of propellants. The test fixture is a stainless steel cylinder, 0.22 inches in diameter by 1-1/2 inches long, and is closed at the bottom with a shielded thermocouple. The fixture is charged with 0.5 cm^3 of sample and closed at the top with a 0.003 inch thick stainless steel diaphragm. Because the NOS-365 reacted violently under this confinement, the stainless steel diaphragm was replaced with an aluminum foil disc to permit earlier release of high pressures that may develop. The assembly was placed in a water bath and heated at a constant rate of 10 C/minute. A second

thermocouple and an X-Y recorder are connected with the sample thermocouple to yield a plot of the differential temperature (sample temperature minus bath temperature) versus bath temperature. Exothermic reactions appear as positive peaks; while, endothermic reactions are negative peaks. Results are reported as the onset temperature at which significant thermal activity first appeared, and the temperature at which catastrophic decomposition occurred (See Appendix A).

Results of Sensitivity Experiments

Task 1: Trauzl Block Tests:

The test results for the Trauzl Block are presented in Table 2. As shown in the table, all the grease/NOS-365 mixtures gave an expanded volume of 1.0 cm³/g or less. When compared to the expanded volume for the test with neat NOS-365 (1.0 cm³/g), one can state that no detectable reaction occurred. Molykote 33/LP 1845 and Molykote FS 3451/LP 1845 mixtures produced a slight increase in the volume (0.5-1.0 cm³/g) when compared to neat LP 1845 (0 cm³/g). However, Molykote M-77 paste/LP 1845 and Fel-Pro C100/LP 1845 mixtures were more sensitive (1.5-2.0 cm³/g).

Task 2: JANAF Thermal Stability Tests

The results of these tests are summarized in Table 3 and shown in Appendix A. As shown in this table, the onset temperature for neat NOS-365 (112 C) was lowered appreciably by the Molykote M-77 paste, Fel-Pro C100, and Molykote FS 3451 grease/NOS-365 mixtures (68-85 C). The Molykote 33/NOS-365 mixture did not lower the onset temperature. This same pattern was observed in comparing the temperatures of the thermal explosion.

The onset temperature for neat LP 1845 (96 C) was not lowered significantly by the Molykote M-77 grease/LP 1845 mixture (95 C). Molykote FS 3451 and Molykote 33 mixtures gave onset temperatures of 80 and 82 C, respectively; while, the most severe change occurred with Fel-Pro C100 whose onset temperature was 76 C. The Molykote M-77 paste/NOS-365 mixture did not appear to effect the onset temperature. When comparing the effects upon the temperatures for the thermal explosion of neat NOS-365 (96 C) and the grease/NOS-365 mixtures, Molykote 33, Molykote FS 3451, and Molykote M-77 paste had little change effect; however, Fel-Pro C100 reduced the thermal explosion temperature to 88 C.

CONCLUSIONS

The results of the detonation tests with NOS-365 and LP 1845 packaged in 500 ml Nalgene polyethylene bottles in DOT-12B No. 275 fiber cartons with vermiculite fill indicated that these materials may be shipped as oxidizing/corrosive liquids. Vermiculite contains approximately 5% iron oxide which will react with the HAN-based liquid propellant. This reaction negates the use of vermiculite as a fill material.

All the detonation tests were negative to high order detonation propagation. This was evidenced by the decaying propagation velocity and the failure to produce a hole in the witness plate.

The fractured witness plates in the detonation tests were a result of a substantial amount of energy release from the RDX initiating charge, decomposition of the liquid propellants and hydraulic ram effects produced by the donor shock wave. This is based upon a comparison of the results from the HAN-based liquid propellants and the water/salt tests.

The Trauzl Block test results on the silicone grease mixtures of NOS-365 and LP 1845 were indicative that no major changes in shock sensitivity occurred after conditioning for 24 hours at ambient temperatures. The LP 1845 grease mixtures were, however, more sensitive to shock conditions than NOS-365 grease mixtures.

The results of the thermal stability tests showed that when NOS-365 was mixed with Molykote M-77 paste, Fel-Pro C100, and Molykote FS 3451 greases, the thermal stability was significantly reduced. NOS-365 mixed with Molykote 33 was thermally stable. The addition of Molykote M-77 paste, Molykote 33, and Molykote FS 3451 to LP 1845 does not significantly reduce its thermal stability. Fel-Pro C100 had the greatest effect on reducing the thermal stability of LP 1845.

RECOMMENDATIONS

NOS-365 and LP 1845 packaged in 500 ml polyethylene containers in DOT-12B No. 275 fiber cartons is recommended for shipment as an oxidizer/corrosive liquid.

Molykote 33 is recommended for use with NOS-365. Molykote M-77 paste, Fel-Pro, and Molykote FS 3451 are not recommended for use with NOS-365.

Molykote 33, Molykote M-77 paste, and Molykote FS 3451 greases are recommended for use with LP 1845, Fel-Pro C100 is not recommended for use with LP 1845.

Vermiculite is not preferred as a package fill for liquid propellant shipments. An alternate fill should be used, such as an open-celled polyethylene foam, that is compatible with the liquid propellants, can absorb spilled materials, and act as a cushion from external shocks.

Table 1. Detonation results

<u>Test No.</u>	<u>Description</u>	<u>Velocity (m/sec)</u>	<u>Remarks</u>
1	500-ml PE bottle water	5500/1900 decaying	Plate OK, bottle torn apart
2	500-ml PE bottle LP 1845	4200/2300 decaying	Plate OK, bottle torn apart
3	500-ml PE bottle NOS-365	4400/2500 decaying	Plate OK, bottle torn apart
4	500-ml PE bottle NOS-365 overpacked in DOT-12 NO. 275 (double wall) fiber box; 4 similar packages in cruciform formation	4000/2500 decaying	Plate broken, carton and bottle destroyed, other packages did not detonate
5	Same as 4 except LP 1845	4100/2700 decaying	Plate bowed and broken carton and bottle destroyed other packages did not detonate
6	Same as 4 except salt water (d=1.35 gm/cc)@75C no acceptor units	5700/3500/ 2100	Plate OK, carton and bottle destroyed

PE = polyethylene

Table 2. Trauzl Block test results

<u>Sample Identification</u>	<u>Test 1*</u> <u>(cm³/g)</u>	<u>Test 2*</u> <u>(cm³/g)</u>
NOS-365	1	1
NOS-365/Molykote 33	0	1
NOS-365/Molykote FS 3451	0.5	1
NOS-365/Molykote M-77 paste	1	1
NOS-365/Fel-Pro C100	1	0.5
LP 1845	0	0
LP 1845/Molykote 33	0.5	1
LP 1845/Molykote FS 3451	0.5	1
LP 1845/Molykote M-77 paste	2	2
LP 1845/Fel-Pro C100	1.5	2

* Net expansion of block for 2 gram charge.

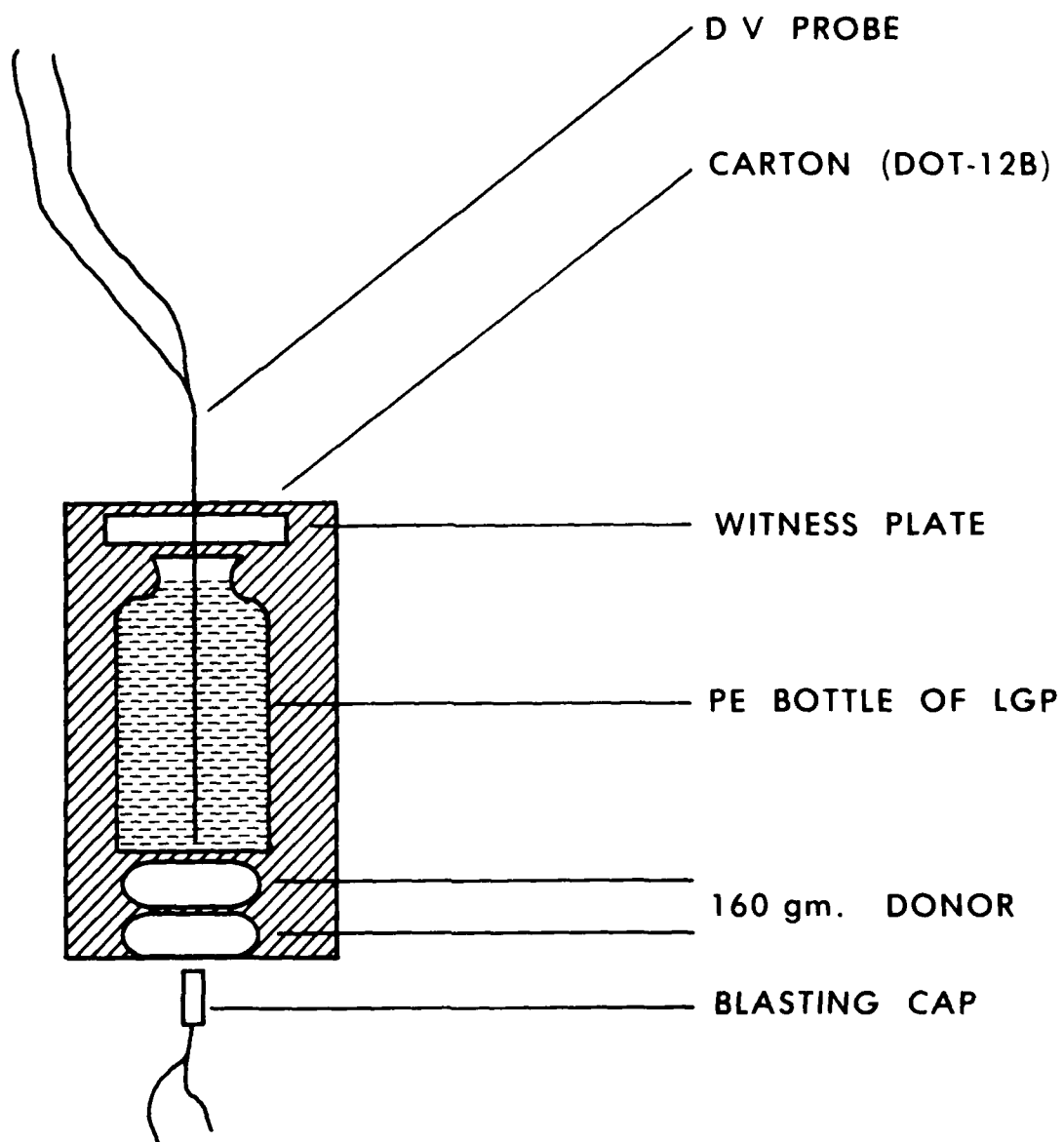
Table 3. JANAF thermal stability test results

Test No.	Sample Identification ¹	Temp of onset of self-heating (°C)	Temp. of Thermal Expl. (°C)	Remarks ³
1	NOS-365	112	135	Destroyed test vessel
2	NOS-365/Molykote 33	120	130	Ruptured burst disc
3	NOS-365/Molykote FS 3451	85	85	Same as 2
4	NOS-365/Molykote M-77 paste	68	68	Same as 2
5	NOS-365/Fel-Pro C100	70	80	Same as 2
6	LP 1845 ²	96	96	Same as 2
7	LP 1845/Molykote 33	80	95	Same as 2
8	LP 1845/Molykote FS 3451	82	92	Same as 2
9	LP 1845/Molykote M-77 paste	95	95	Same as 2
10	LP 1845/Fel-Pro C100	76	88	Same as 2

1. All mixtures were premixed in equal quantity weights. Normal charge weight was 0.50 grams.

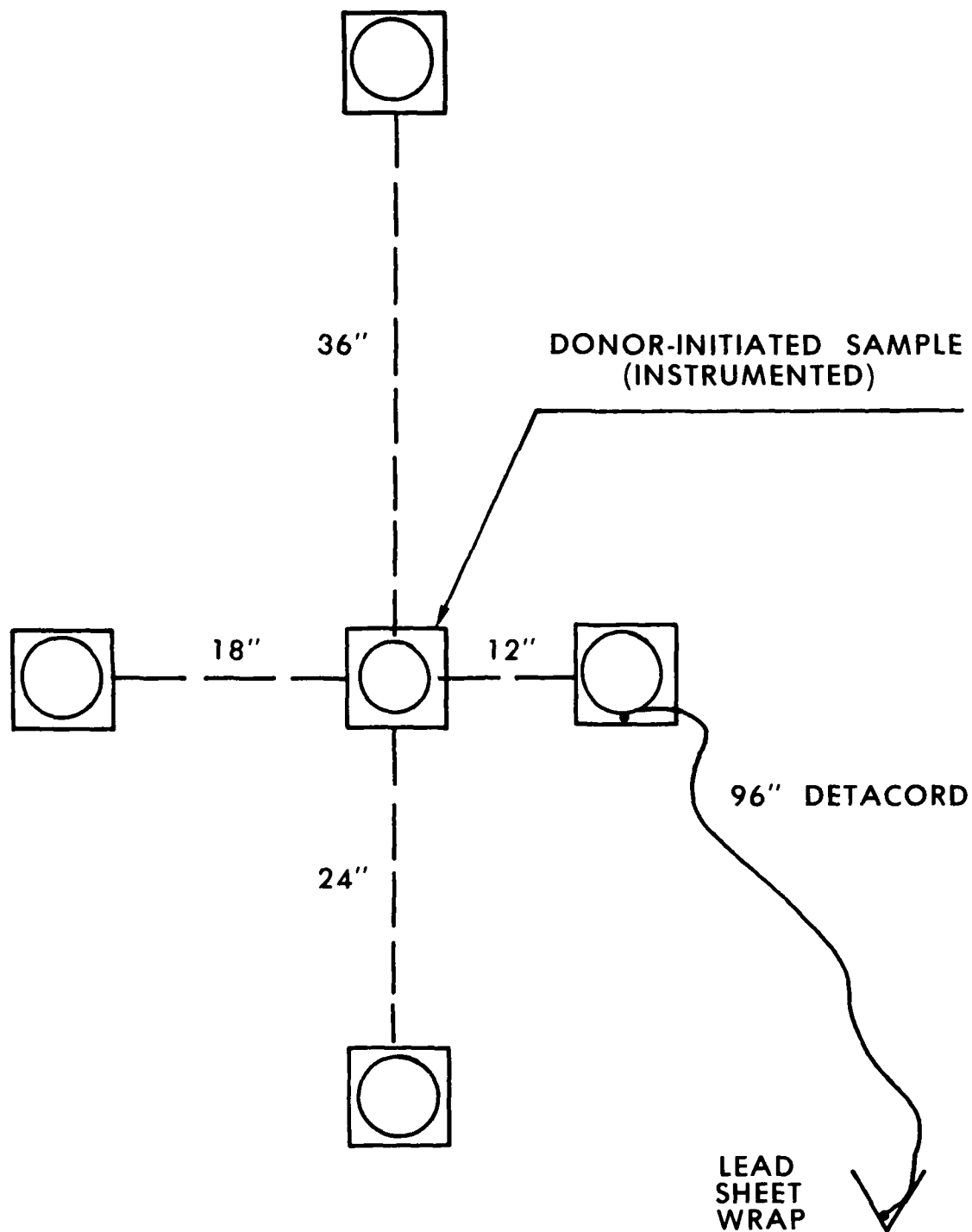
2. Sample charge weight was 0.25 grams.

3. Except for initial trial with NOS-365, burst disc was a thin piece of aluminum foil, no burst rating was determined. All reactions were very rapid.



NOTE:
SHADED AREA OCCUPIED BY COMMERCIAL VERMICULITE.

Figure 1. Cross-section schematic of liquid propellant sample for detonation test



NOTE: ALL PE BOTTLES IN FIBER CARTONS WITH VERMICULITE

Figure 2. Schematic of test array



Figure 2a. Actual test setup for detonation test

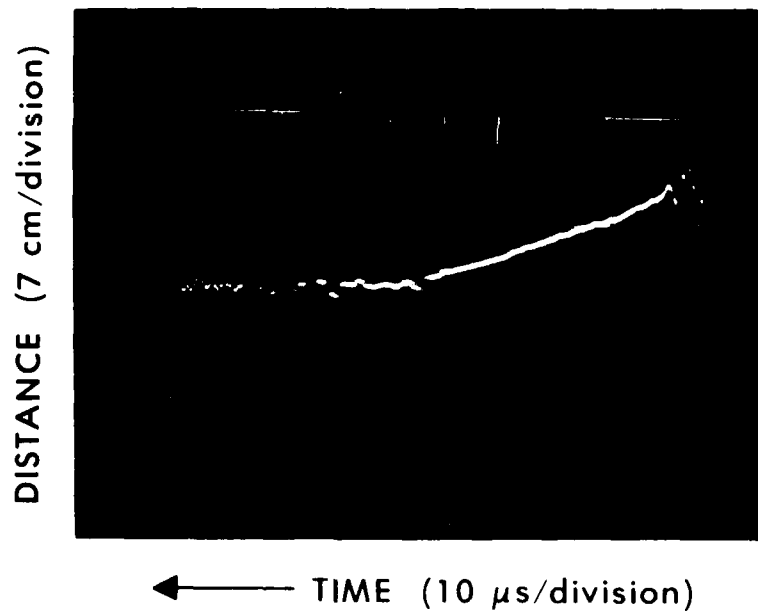


Figure 3. Distance-time trace of detonation test for water

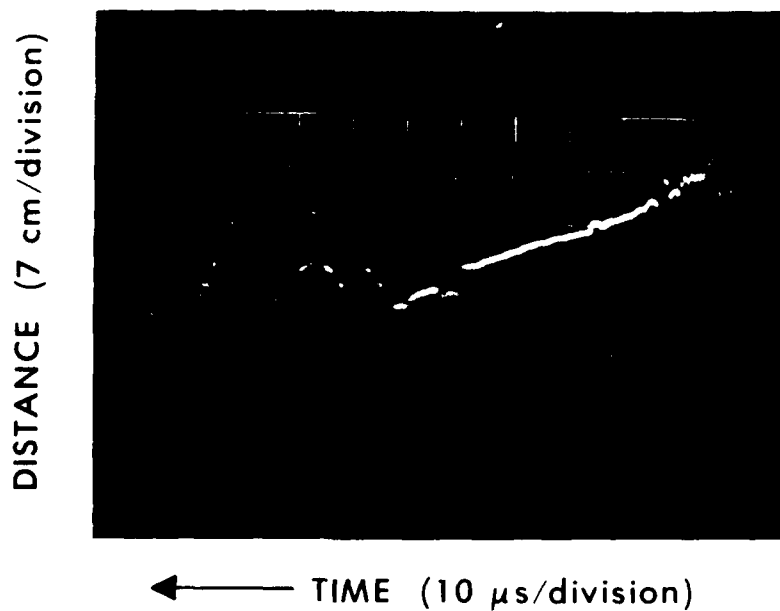


Figure 4. Distance-time trace of detonation test for 1.34 density salt/water mixture

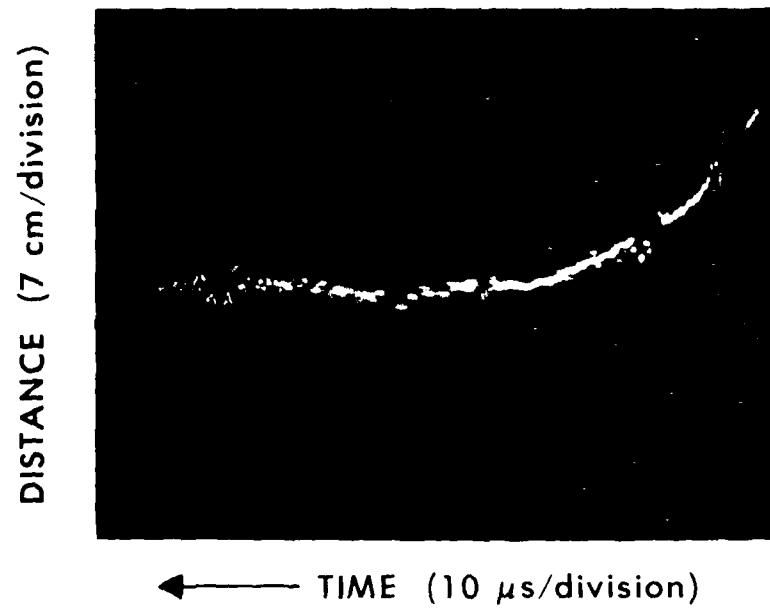


Figure 5. Distance-time trace of detonation test for NOS-365

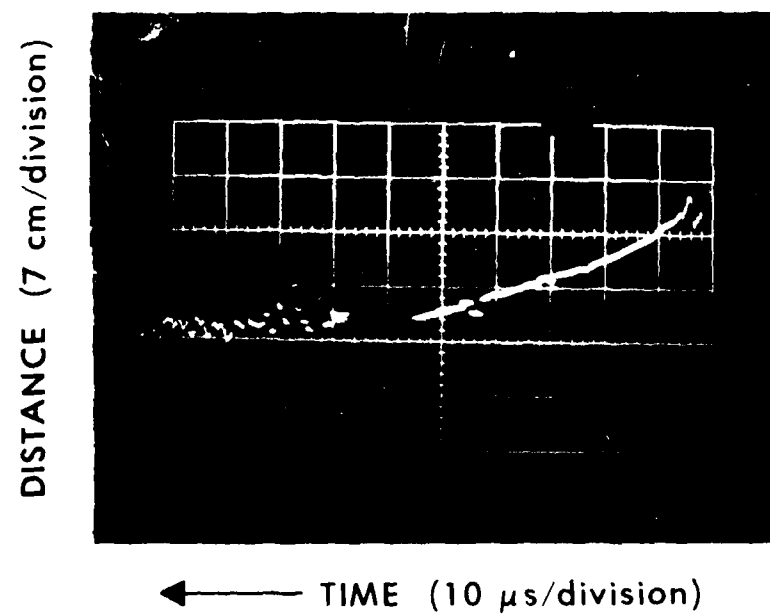


Figure 6. Distance-time trace of detonation test for LP 1845

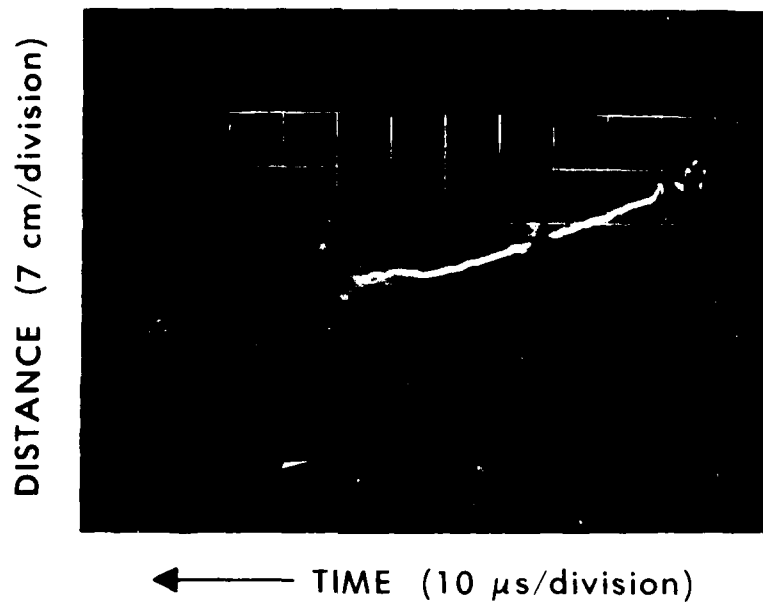


Figure 7. Distance-time trace of detonation test for NOS-365 array

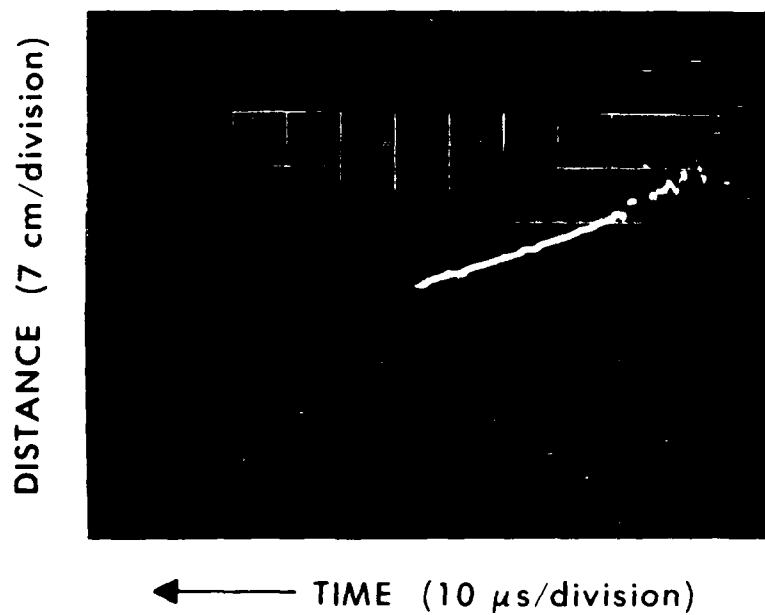


Figure 8. Distance-time trace of detonation test for LP 1845 array



Figure 9. Detonation test results for NOS-365 array

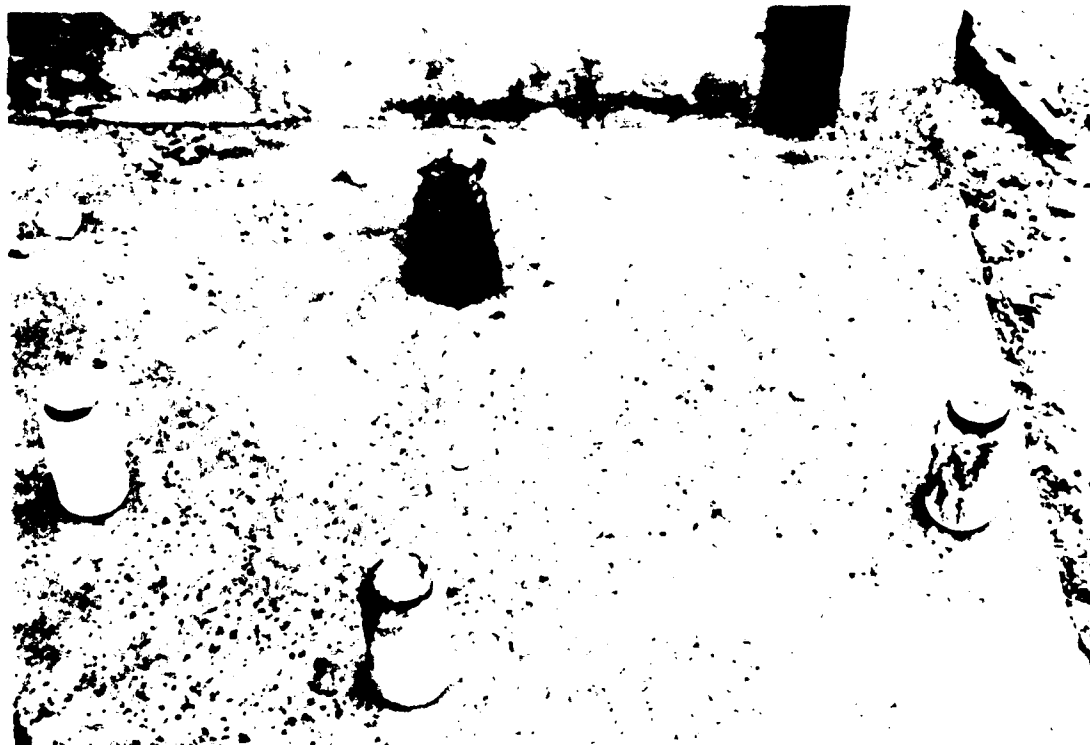


Figure 10. Detonation test results for LP 1845 array

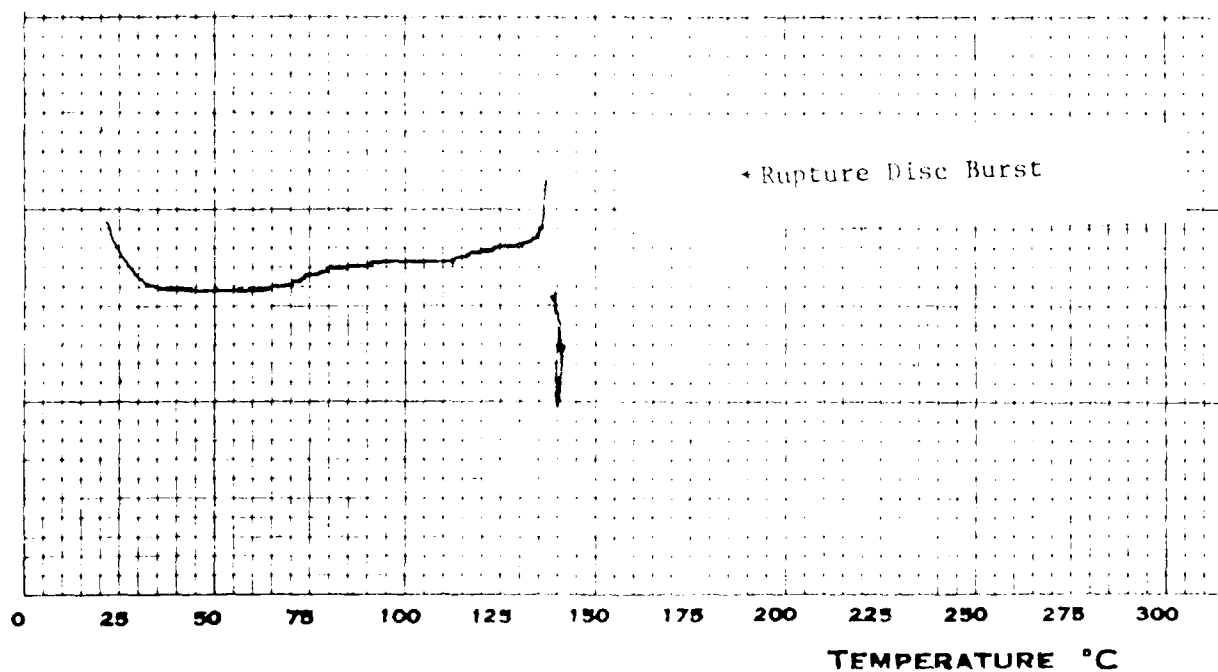


Figure 11. Witness plate from NOS-365 detonation test

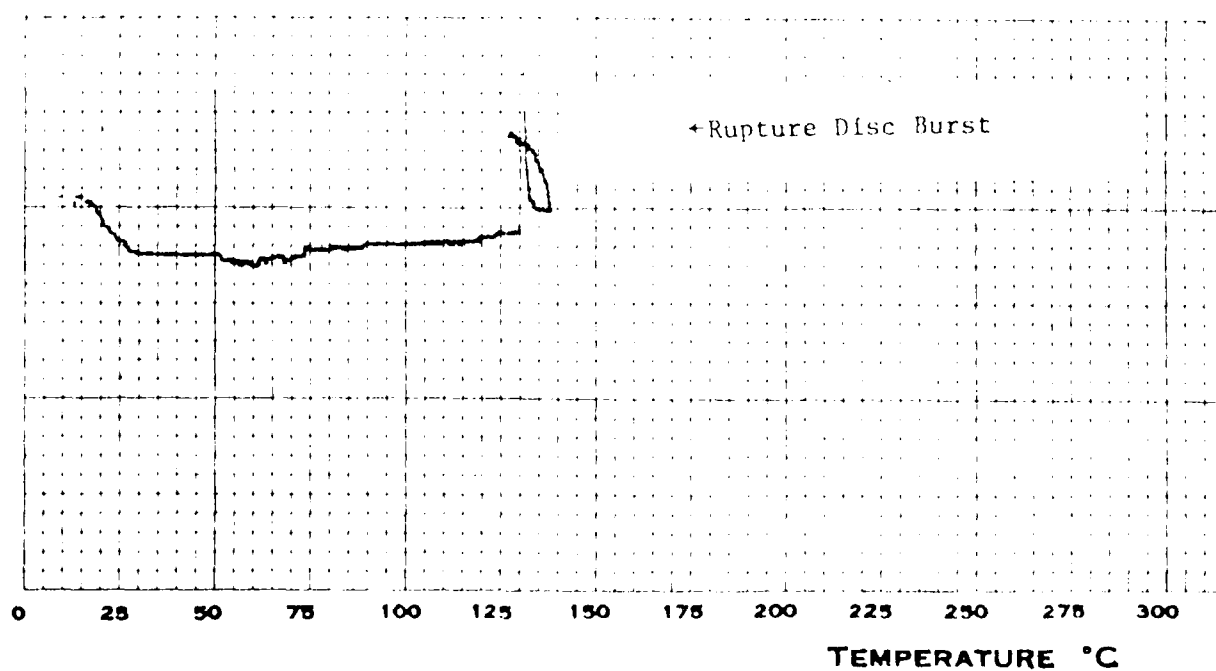


Figure 12. Witness plate from LP 1845 detonation test

APPENDIX
THERMAL ACTIVITY

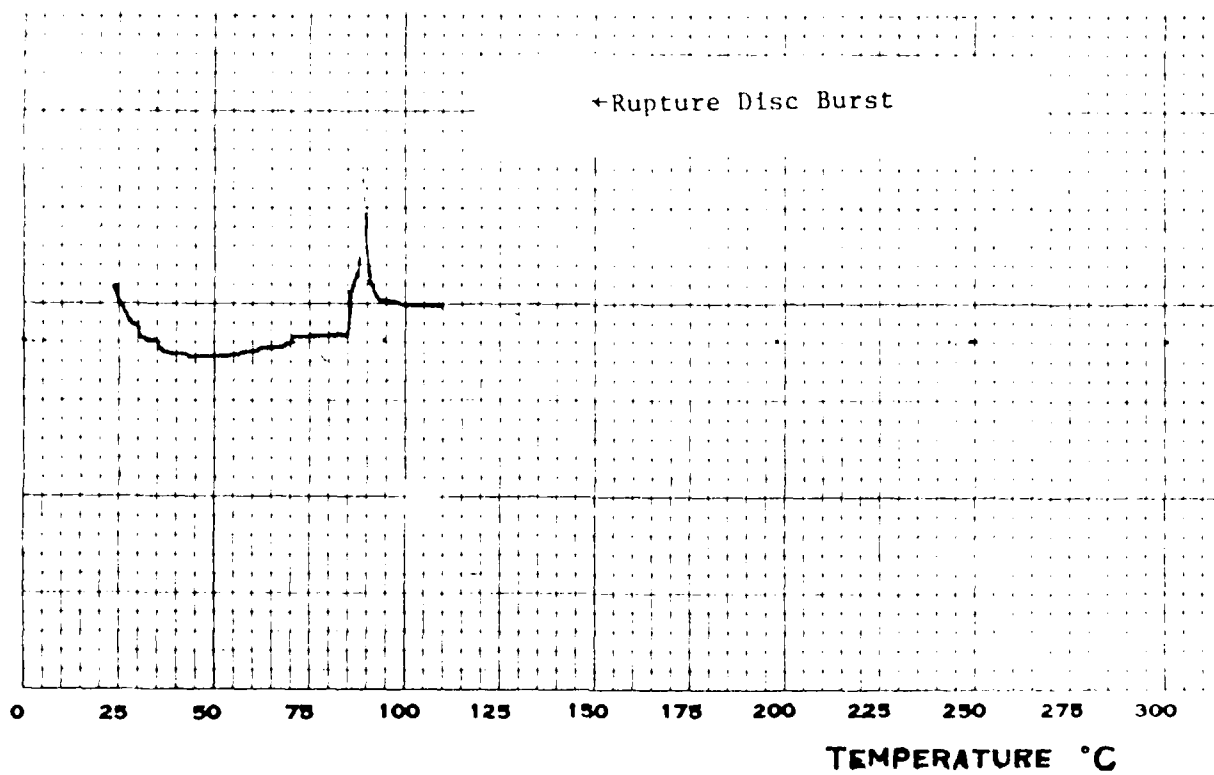


Run 1, .5 grams, NOS-365

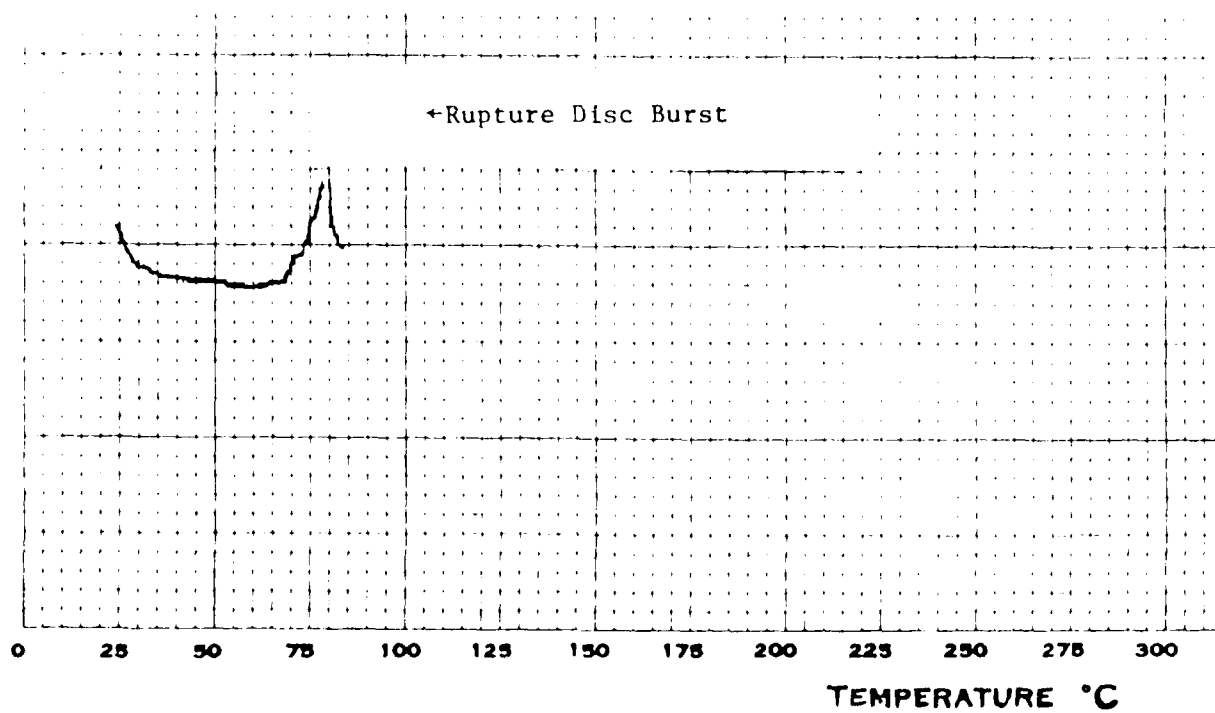


Run 1a, .5 grams, NOS-365 + Molykote 33

Figure A-1. JANAF thermal stability--rupture disc burst

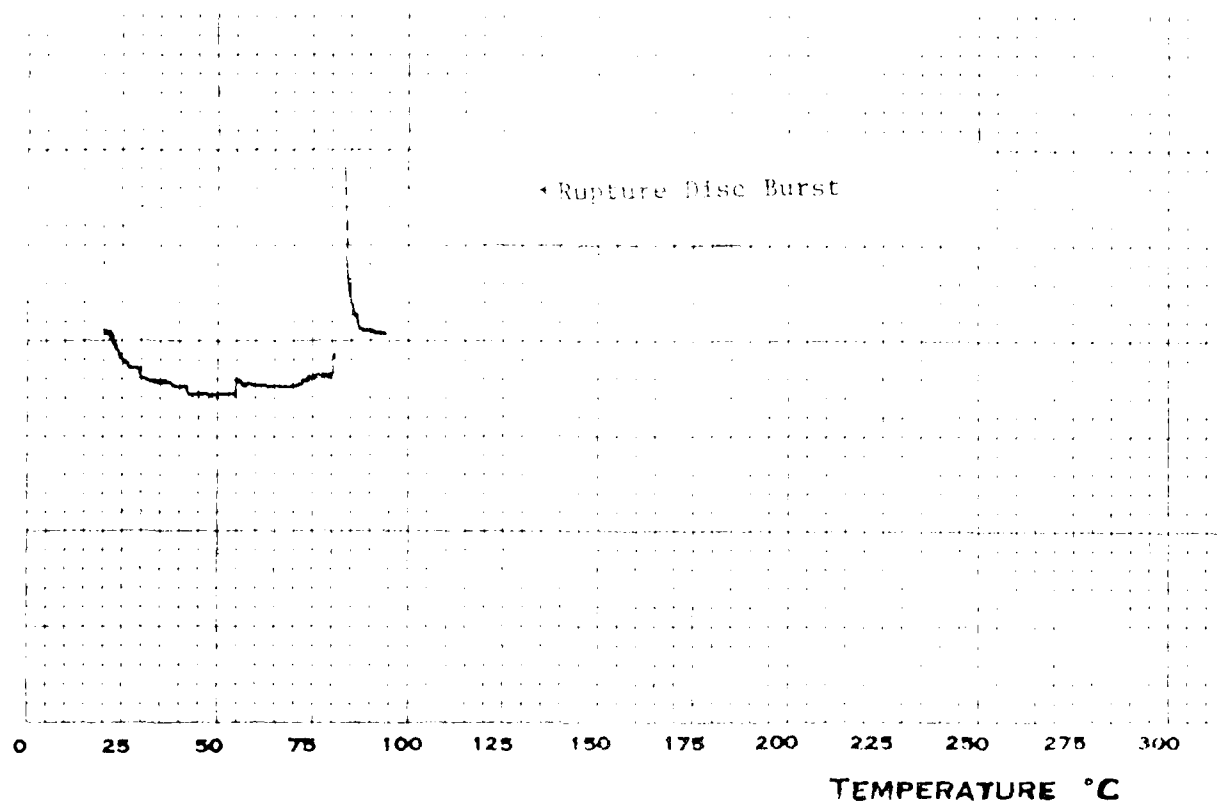


Run 1b, .5 grams, NOS-365 + FS-3451

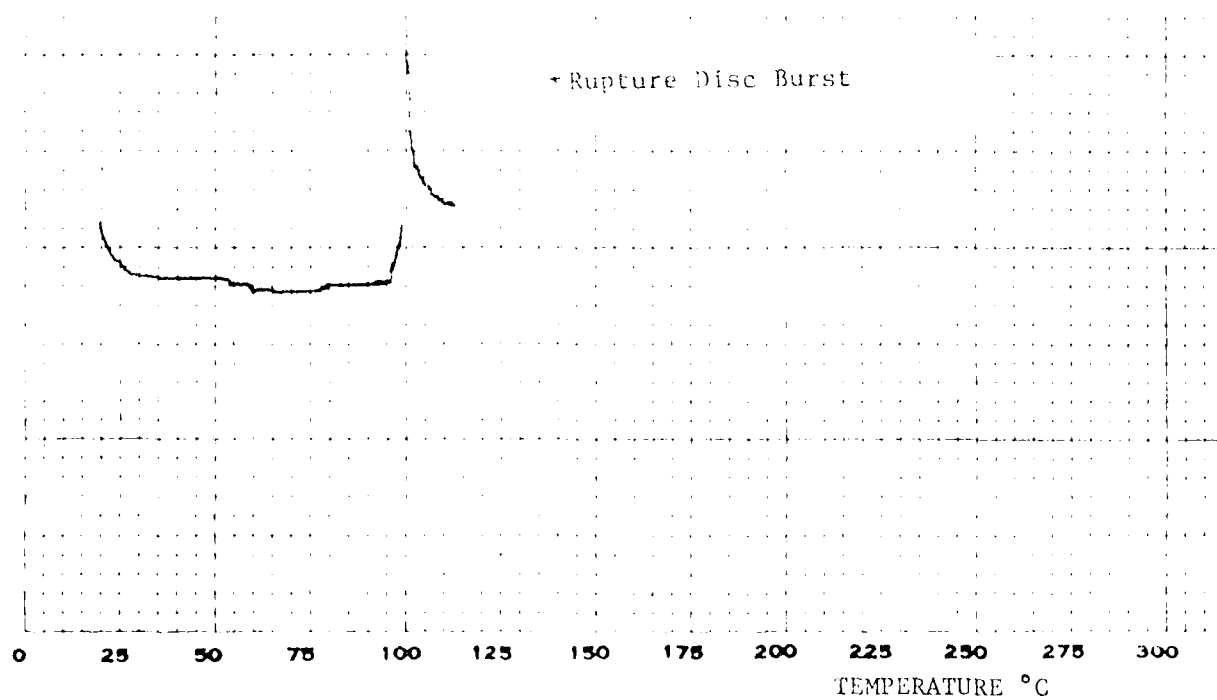


Run 1c, .5 grams, NOS-365 + M-77 paste

Figure A-1. (cont)

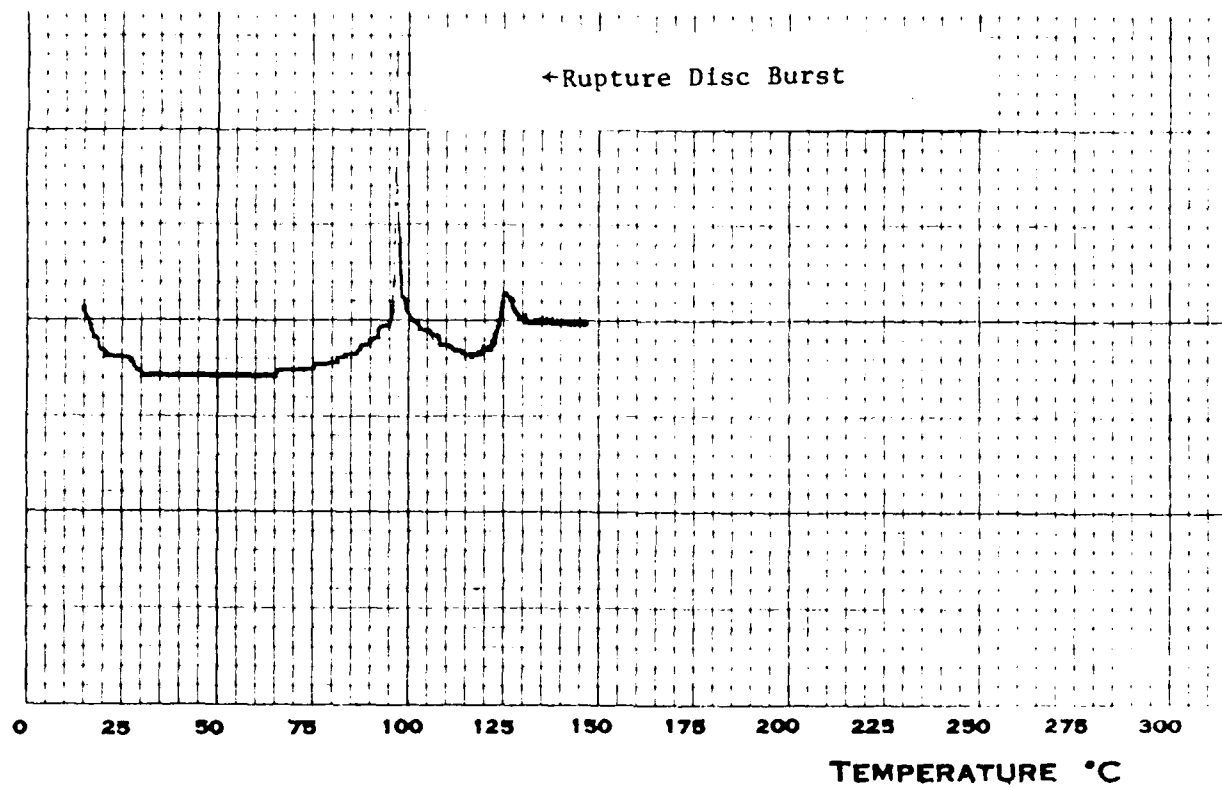


Run 1d, 1.0 gram, N₂O-365 + Fel-Pro

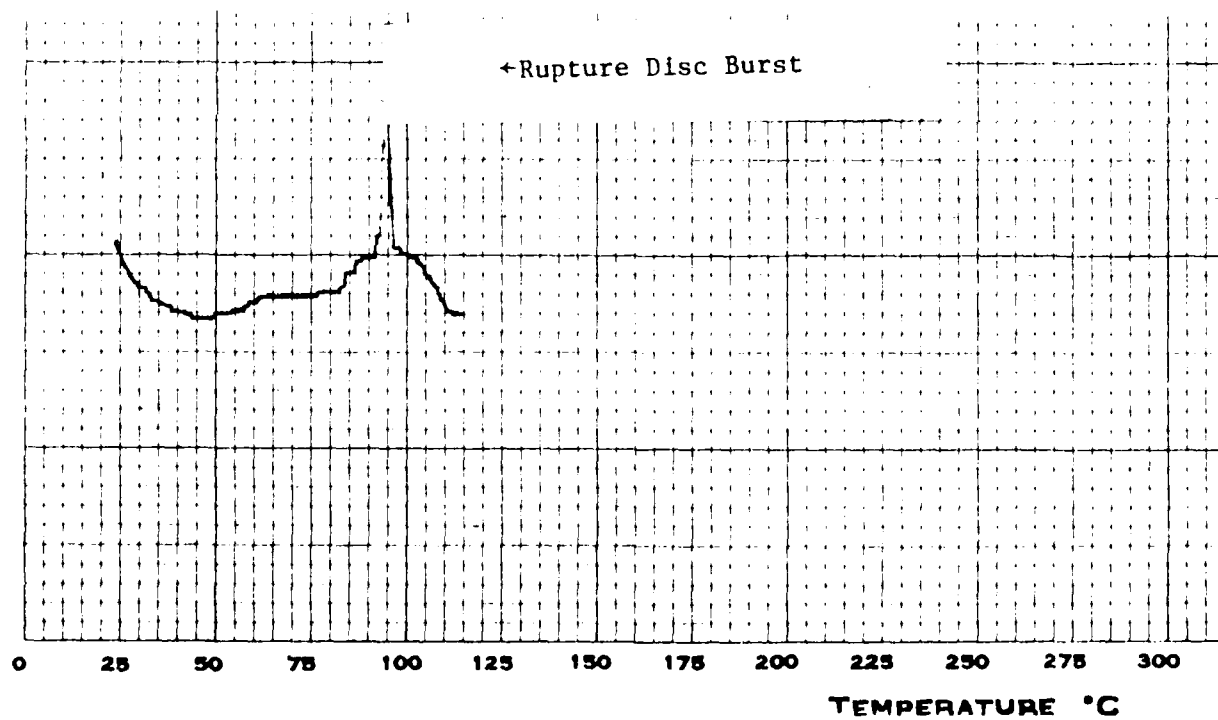


Run 2, 1.0 gram, IP 1845

Figure A-1. (cont)

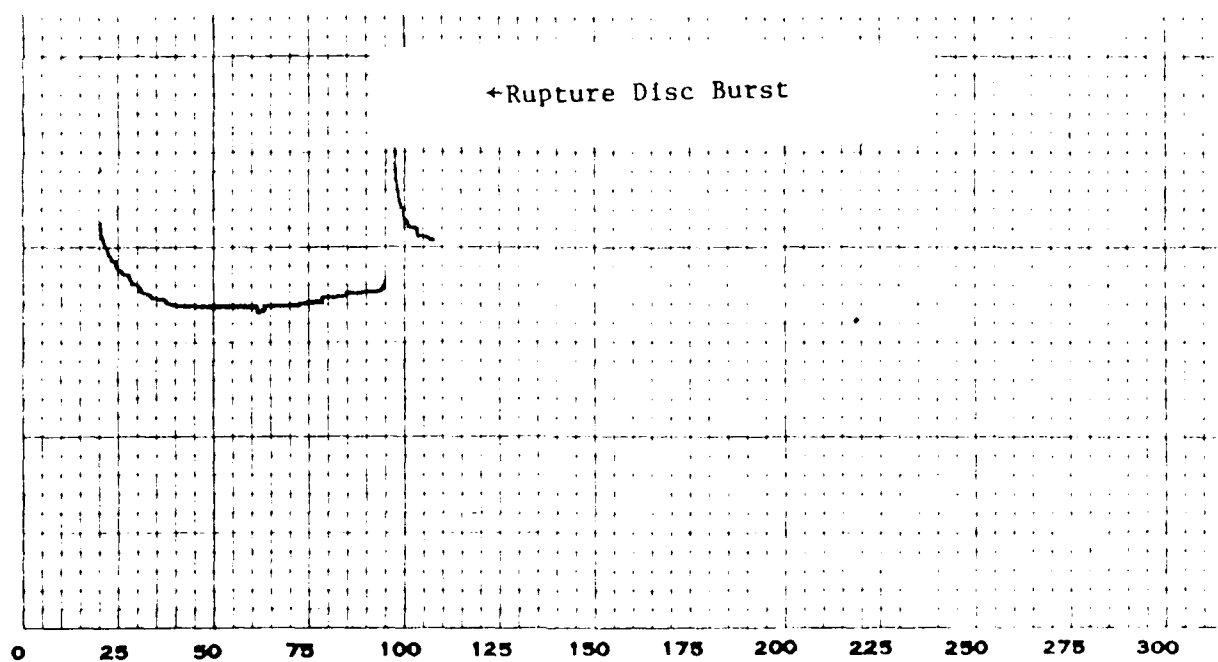


Run 2a, .5 grams, LP 1845 + Molykote 33

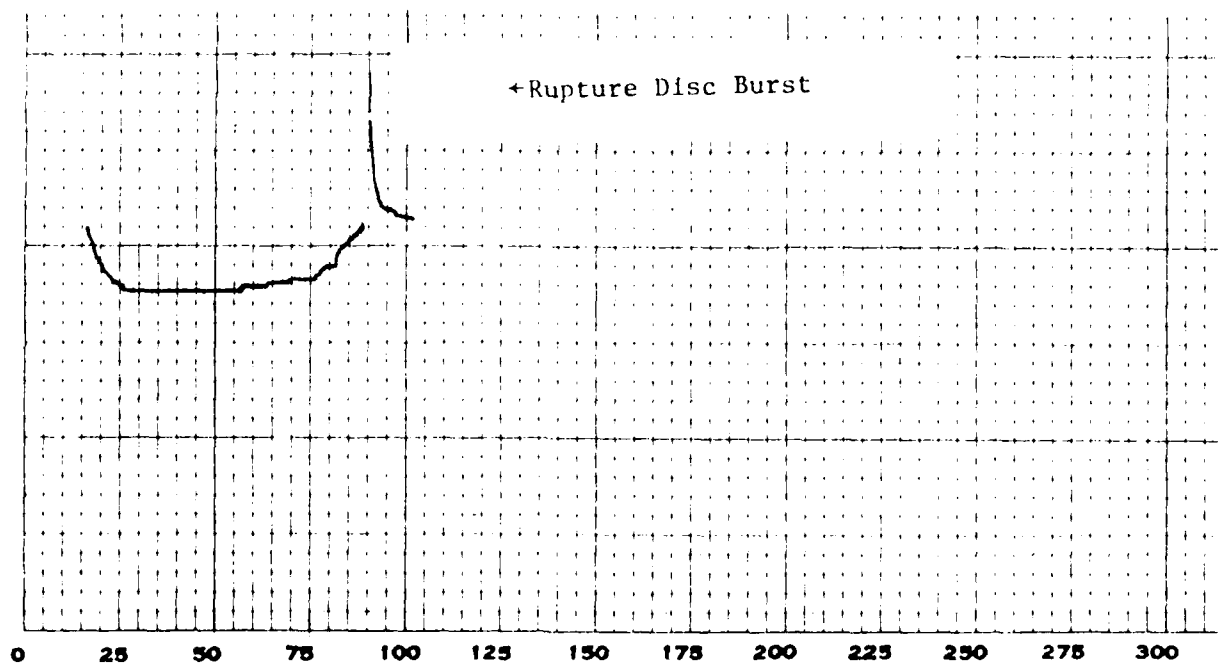


Run 2b, .5 grams, LP 1845 + FS 3451

Figure A-1. (cont)



Run 2c, .5 grams, LP 1845 + M-77 paste



Run 2d, .5 grams, LP 1845 + Fel-Pro

Figure A-1. (cont)

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U.S. Army Armament, Munitions
and Chemical Command
ATTN: AMSMC-GCL(D)
Dover, NJ 07801-5001

Administrator
Defense Technical Information Center
ATTN: Accessions Division (12)
Cameron Station
Alexandria, VA 22304-6145

Director
U.S. Army Materiel Systems
Analysis Activity
ATTN: AMXSY-MP
Aberdeen Proving Ground, MD 21005-5066

Commander
Chemical Research and Development Center
U.S. Army Armament, Munitions
and Chemical Command
ATTN: SMCCR-SPS-IL
Aberdeen Proving Ground, MD 21010-5423

Commander
Chemical Research and Development Center
U.S. Army Armament, Munitions
and Chemical Command
ATTN: SMCCR-RSP-A
Aberdeen Proving Ground, MD 21010-5423

Director
Ballistic Research Laboratory
ATTN: AMXBR-OD-ST
AMXBR-IBD, Charles Leveritt
Aberdeen Proving Ground, MD 21005-5066

Chief
Benet Weapons Laboratory, CCAC
Armament Research, Development and
Engineering Center
U.S. Army Armament, Munitions
and Chemical Command
ATTN: SMCAR-CCB-TL
Watervliet, NY 12189-5000

Commander
U.S. Army Armament, Munitions
and Chemical Command
ATTN: SMCAR-ESP-L
Rock Island, IL 61299-6000

Director
U.S. Army TRADOC Systems
Analysis Activity
ATTN: ATAA-SL
White Sands Missile Range, NM 88002

Hazards Research Corporation
ATTN: William J. Cruice (2)
200 E. Main Street
Rockaway, NJ 07866

END

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